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(54) IMPROVEMENTS IN OR RELATING TO CAPILLARY VISCOMETERS

(71) We, SPETSIALNOE KONSTRUKTORSKOE BJURO INSTITUTA NEFTEKHIMICHESKOGO SINTEZA IMENI A.V. TOPCHIEVA AKADEMII NAUK SSSR, of Leninsky Prospekt 31, Moscow, Union of Soviet Socialist Republics, a corporation organised and existing under the laws of the Union of Soviet Socialist Republics, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to capillary viscometers. Such capillary viscometers may be used in investigating viscous properties of disperse and polymeric systems of a material to be investigated, and based on the flow curve principle, the flow curve showing the rate of shear versus shearing stress.

There are known viscometers, wherein the force of a preliminary compressed spring is imparted to a piston capable of forcing material to be investigated through a capillary and whose displacement is recorded by recording means adapted for the purpose.

In these devices in order to obtain the dependence of the viscosity of the material to be investigated on the pressure drop on the capillary and on the rate of shear use is made of a calibrated spring, which imparts the pressure varying in the process of taking measurements to the material to be investigated, thereby providing a varying rate of efflux of said material from the capillary.

The displacement of the piston forcing out the material from the chamber is recorded, by the recording means adapted for the purpose, with time, with the result that both the shear rate and stress are obtained. However, the thus-recorded data need to be decoded, i.e., graphical differentiating and taking logarithms are necessary to present the results in a form suitable for analysis because the rate of shear versus the shearing stress relationship generally uses a log-log scale.

Besides, when using said viscometers, manual recording rate switching is required due to the kind of the curve being recorded. In some cases, this is impractical and, when rapidly varying rates of recording are involved, it is not always possible.

The present invention consists in a capillary viscometer comprising a spring, means for preliminary compressing the spring, the spring being arranged to act on a piston so as, in use, to force material to be investigated out of a chamber through a capillary, a transducer for converting the displacement of the piston into an output signal, and means for converting the output signal into a voltage proportional to the common logarithm of the rate of displacement of the piston and into a voltage proportonal to the common logarithm of the pressure created by the spring, the converting means being connected to a recording means for recording the size of the voltages.

It is advantageous that the transducer comprises an optical grating-type transducer.

Preferably, the means for converting the output signal from the transducer comprises a digital meter connected to the transducer for recording the rate of displacement of the piston, a code converted connected to the outputs of the meter and to an input of the recording means for converting the code from the digital meter into a voltage proportional to the common logarithm of the rate of dis-

placement of the piston, a counter for counting pulses proportional in number to the displacement of the piston and connected to the transducer, and a code converter for converting the code from the counter into a voltage proportional to the common logarithm of the pressure created by the spring, connected to the outputs of the counter and to another input of the recording means, the recording means being arranged to produce a representation of a flow curve on its chart tape.

Such an embodiment of the herein-proposed capillary viscometer enables presentation of the viscosity of the material to be investigated, within a wide range of values of the rate of shear and the shearing stress (with the maximum-to-minimum shearing stress ratio of 10^2, and maximum-to-minimum rate of shear ratio of 10^5) without additional arithmetic calculations, as a flow curve in a desired logarithmic scale along the X-axis and Y-axis.

The invention will be further described, by way of example only, with reference to the accompanying drawings, wherein:

Fig. 1 is a longitudinal cross-sectional view of a preferred capillary viscometer;

Fig. 2 is a block diagram of means for converting the output signal of the capillary viscometer transducer of Fig. 1;

Fig. 3 is a section taken along the line A of Fig. 1 in an enlarged scale; and

Fig. 4 is a flow curve obtained by means of the capillary viscometer of Fig. 1.

With reference to Fig. 1, the capillary viscometer incorporates a chamber 1, part of which is filled with a material 2 to be investigated which is forced out through a calibrated cylindrical capillary 3 of the chamber 1 by the force of a preliminarily compressed spring 4, the force being imparted to a piston 5 on whose rod 6 the spring 4 is fixed in position.

The viscometer also incorporates a transducer 7 for converting discrete displacement of the piston 5, operatively associated with the rod 6, and a means 8 (Fig. 2), electrically connected to the transducer 7, capable of converting the output signal of the transducer 7 into a voltage proportional to the common logarithm of the rate of displacement of the piston 5 (Fig. 1) and to the common logarithm of the pressure created by the spring 4.

As the transducer 7 is an optical grating-type transducer comprising two transparent plates 9 and 10 with a uniform linear grating 11 (Fig. 3) traced thereon; the plate 10 (Fig. 1) is connected to, and hence movable with, the rod 6 of the piston 5, while the other plate 9 is fixed to a casing 16. The optical grating-type transducer further comprises a light source 12 in the form of a filament lamp whose light beam (in the directions indicated by the arrows B) is incident upon the plate 9, passes therethrough and through the plate 10, and is fed (in the direction indicated by the arrows C) to a photoreceiver 13.

The use of a grating-type transducer as the transducer 7 to convert discrete displacement of the piston 5 is necessitated by the high degree of accuracy required in the measurement of the rate of shear and the shearing stress as a result of the use of a chamber 1 of small length. The small length of the chamber 1 allows a small amount of the material 2 to be investigated to be used.

When a greater amount of the material to be investigated is available, the discrete displacement transducer may comprise an inductive transducer provided with a toothed movable armature and a toothed pole tip.

In order for the spring 4 to be compressed in the present viscometer, there is provided a lifting means incorporating a screw 14 placed within the threaded portion of a flywheel 15 rotatably mounted in the casing 16. The screw 14 is provided with a collet grip 17 having a cylinder 18, operatively associated with a releasing means 19 and an upper cap 20 which the upper end of the spring 4 thrusts against. The collet grip 17 engages the upper end face of the rod 6 which is provided with a cap 21 wherein the lower end of the spring 4 rests and the movable plate 10 is mounted.

The means 8 (Fig. 2) for converting the output signal of the discrete displacement transducer 7 (Fig. 1) into voltages proportional to the common logarithm of the rate of displacement of the piston 5 and to the logarithm of the pressure created by the spring 4, incorporates a digital meter 22 (Fig. 2) for determining the rate of displacement of the piston 5 (Fig. 1) connected to the photoreceiver 13 of the discrete displacement transducer 7, and a code converter 23 (Fig. 2) for converting the code available from said meter 22 into a voltage proportional to the logarithm of the rate of displacement of the piston 5 (Fig. 1) and which is connected to the outputs of the meter 22 (Fig. 2) and to the input "Y", corresponding to the coordinate "Y", of a recording means 24 so as to record the rate of shear. The recording means 24 is a two-coordinate recorder. The means 8 for converting the output signal of the discrete displacement transducer 7 (Fig. 1) further comprises a counter 25 (Fig. 2) for determining the displacement of the piston 5 (Fig. 1), connected to the photoreceiver 13 of the discrete displacement transducer 7, and a code converter 26 (Fig. 2) for converting the code from the counter 25 into a voltage proportional to the logarithm of the pressure created by the spring 4 (Fig. 1) and which is connected to the outputs of the counter 25 (Fig. 2) and to the input "X", corresponding to the coordinate "X", of the

recording means 24 so as to record the shearing stress. Thus, the chart tape 27 (Fig. 4) of the recording means 24 (Fig. 2) provides a flow curve 28 (Fig. 4), whereon the values of the shearing stress τ (in dn/cm^2) and those of the rate of shear D (in sec^{-1}) are plotted on a logarithmic scale along the X-axis and the Y-axis, respectively.

The digital meter 22 (Fig. 2) for determining the rate of displacement of the piston 5 (Fig. 1) incorporates a key switch 29 (Fig. 2), one of whose inputs is connected to the photoreceiver 13, whereas its other input is connected to one of the reference pulse generators 30*k*, 30*l*, 30*m*, 30*n* or 30*p* by means of a step selector switch 31. The number of reference pulse generators is chosen to suit the number of the measurement subranges with respect to rate which make up the entire range of measurement of the viscometer, according to the present invention. The herein-proposed embodiment features five measurement subranges.

The output of the key switch 29 is electrically connected to a counter 32, each stage of which is connected to a storage means 33.

The digital meter 22 also comprises circuits for comparing the contents of the counter 32 with a minimum value 34 and a maximum value 35. These circuits are essentially "AND" circuits with a number of inputs equal to the number of digits of the counter 32, and trigger circuits 36 and 37, the inputs of the "AND" circuits 34 and 35 being connected to those stages of the counter 32 necessary to determine when the contents thereof corresponds to the beginning ("AND" circuit 34) and end the (the "AND" circuit 35) of each measurement subrange respectively, and the outputs being connected to the trigger circuits 36 and 37 respectively. The outputs of the trigger circuits 36 and 37 are connected to the four inputs of a control decoder 38 whose fifth input is connected to the output of the photoreceiver 13.

The control decoder 38 has four outputs, two of which are connected to the step selector switch 31, the third of which is connected to the storage means 33, and the fourth to the counter 32 and the triggers 36 and 37.

The code converter 23 to convert the code available from the digital rate meter 22 into the voltage proportional to the common logarithm of the rate of displacement of the piston 5 (Figure 1) incorporates a mantissa digital-to-analog converter 39 (Figure 2), connected to the storage means 33 of the rate meter 22, a logarithmic converter 40, connected to the converter 39, an adder 41, one of whose inputs is connected to the converter 40 and the other to a charcteristic digtal-to-analog converter 42. The characteristic digital-to-analog converter 42 is connected to the step selector switch 31 of the digital rate meter 22.

The number of the channels between the mantissa digital-to-analog converter 39 and the storage means 33 of the digtal rate meter 22 and between the characteristic digital-to-analog converter 42 and the step selector switch 31 is arranged to suit the number of the digits of the counter 32 (in the herein-proposed embodiment there are eight digits), each of whose digits are connected *via* the storage means to the respective input of the digital-to-analog converter 39, and the number of steps of the step selector switch 31, which is due to the number of the subranges (five) of the digital rate meter 22 respectively.

The output of the adder 41 is connected to the "Y" input of the recording means 24.

The code converter 26 for converting the code available from the counter 25 into a voltage proportional to the logarithm of the pressure created by the spring 4 (Fig. 1), incorporates a digital-to-analog converter 43 (Fig. 2), connected to the outputs of the counter 25, and a logarithmic converter 44, connected to the output of the digital-to-analog converter 43. The number of the channels between the counter 25 and the converter 43 is arranged to suit the number of the digits of the counter 25 (there are eight digits in the herein-proposed embodiment).

The output of the functional converter 44 is connected to the "X" input of the recording means 24.

The operating principle of the present capillary viscometer is as follows.

Prior to starting measurements, the spring 4 is compressed by rotating the flywheel 15 (Fig. 1) of the lifting means so that the upper end face of the rod 6 is gripped by the collet grip 17. Using the releasing means 19, the collet grip 17 is fixed by lowering the cylinder 18. By rotating the flywheel 15 the rod 6 is then lifted to its upper position and the chamber 1 together with the capillary 3 filled with the material 2 to be investigated, is set in position.

When starting measurements, the collet grip 17 is opened by using the releasing means 19 and lifting the cylinder 18, thereby releasing the spring 4. The spring 4 is released to push the piston 5 and the rod 6 so as to force out the charge of the material 2 to be investigated from the chamber 1 through the capillary 3. As the spring 4 is being released, the pressure difference effective in the capillary 3, wherethrough the material to be investigated runs, is decreased with subsequently reduce rate of efflux thereof and, consequently, the rate of displacement of the piston 5 is also reduced. The values of the shearing stress and the rate of shear being measured are determined by the displacement of the spring 4 from its initial position and

by the rate of displacement of the piston 5, respectively.

To determine the above-mentioned values, the piston 5 is connected to the discrete displacement transducer 7. When the piston 5 moves together with said transducer, the movable plate 10 of the transducer 7, which is rigidly connected to the rod 8, also moves. Since the plate 10 moves in the direction perpendicular to the lines of the optical grating 11 (Fig. 3), there occurs a periodic modulation of the light beam coming from the light source 12 (Fig. 1) through the stationary plate 9 and movable plate 10 to the photoreceiver 13 of the transducer 7. This leads to the appearance of a periodic voltage in the photoreceiver 13 which, due to a limited current value in the photoreceiver 13, is converted into a train of pulses whose period is related to the reciprocal of the rate of displacement of the piston 5 and to the width of the lines on the optical grating 11 (Fig. 3), while the total number of the pulses depends on the displacement of the piston 5 (Fig. 1) from its initial position, i.e., on the length of run of the spring 4, and on the spacing between the lines of the optical grating 11 (Fig. 3). The above-mentioned pulses are fed to the input of the key switch 29 (Fig. 2), which receives a signal from one of the reference pulse generators 30*k*, 30*l*, 30*m*, 30*n* or 30*p* *via* the step selector switch 31. This results in the output of the key switch 29 comprising a burst of pulses whose duration is equal to that of the pulse fed from the photoreceiver 13, while the repetition rate of the pulses in the bundle is equal to the frequency of the signal coming from the operating reference pulse generator 30*k*, 30*l*, 30*m*, 30*n* or 30*p*. The number of pulses in the bundle which defines the reciprocal of the rate of displacement of the piston 5 (Fig. 1) is counted by the counter 32 (Fig. 2). Upon termination of the pulse from the photoreceiver 13, the feeding of code from the counter 32 to the storage means is actuated by the trailing edge of the signal from the photoreceiver 13 which is fed from the decoder 38 into the storage means 33.

The code stored in the storage means 33 is converted, by means of the mantissa digital-to-analog converter 39, into a voltage proportional to said code and then, by means of the converter 40, into a voltage proportional to the logarithmic mantissa of the reciprocal of the rate of displacement of the piston 5 (Fig. 1).

To change from one measurement subrange to another the outputs of the counter 32 are connected, according to the extreme values of the codes characterizing the beginning and end of the measurement subrange, with the evaluation circuits 34 and 35 to compare the numbers fed to the counter 32 with the minimum and maximum. If during successive counting it has been found that, by the end of the measurement interval (duration of the pulse coming from the photoreceiver 13), the counter 32 has received N pulses which exceeds the value of N_{min} code set in the evaluation circuit 34, but is less than the value of N_{max} code set in the evaluation circuit 35, then at the moment of reaching N_{min}, the circuit 35 operates and the trigger circuit 36 flips, while the circuit 35 remains in the initial state. This indicates that the measurement subrange and the value of the rate to be measured are in agreement. In this case, upon termination of the measurement subrange, the decoder produces a signal which actuates delivery of the code available from the counter 32 to the storage means 33.

If the number of pulses fed to the counter 32 is found to be less than N_{min} or greater than N_{max}, no flipping of the trigger circuits 36 and 37 takes place or both trigger circuit 36 and 37 are flipped. This being the case and upon termination of the measurement interval, the decoder 38 produces a signal to the step selector switch 31 causing one of the reference pulse generators 30*k*, 30*l*, 30*m*, 30*n* or 30*p* with a higher or lower pulse repetition rate to be connected to the keyswitch. Such switching of the generators will continue until the rate of repetition of the reference pulses is found to agree with the range of measurement of the rate of displacement of the piston 5 (Fig. 1).

After every measurement, the decoder 38 (Fig. 2) sends a signal to the counter 32 and to the trigger circuits 36 and 37 to reset the counter 32 and said trigger circuits.

To allow for the change of the measurement subrange when plotting the flow curve 28 (Fig. 4), the step selector switch 31 (Fig. 2) supplies a signal to the code converter 23, in particular to the characteristic digital-to-analog converter 42, in the form of a position code, which is converted therein into the voltage proportional to the logarithmic characteristic of the reciprocal value of the rate of displacement of the piston 5. The voltage proportional to the logarithmic characteristic of the rate of displacement of the piston 5 (Fig. 1), obtained in the digital-to-analog converter 42 (Fig. 2), and the voltage proportional to the mantissa thereof, obtained in the functional converter 40, are summed in the adder 41. The summed voltage from the adder 41 is fed to the "Y" input of the recording means 24 to record the logarithm of the reciprocal of the rate of displacement of the piston 5 (Fig. 1).

The pulse signal from the photoreceiver 13 (Fig. 2) is also fed to the counter 25 whereinas the pulses are being fed, the current code value of the displacement of the piston 5 (Fig. 1) is formed. By using the code converter 26 (Fig. 2), said current value is con-

verted into a voltage proportional to the common logarithm of the displacement of the piston 5 (Fig. 1) (the lengths of run of the spring 4 from its initial position) for which purpose the code converter 26 (Fig. 2) incorporates the digital-to-analog converter 43 capable of converting the code into the voltage proportional thereto, said voltage, by means of the logarithmic converter 44, being further converted into a voltage proportional to the logarithm of the displacement of the piston 5 (Fig. 1).

The voltage from the output of the converter 44 (Fig. 2) is fed to the "X" input of the recording means 24 to record the logarithm of the run of the spring 4 (Fig. 1) from its initial position, i.e., the logarithm of the change of pressure from its initial maximum value.

It is well to note that the pressure created by the spring 4 varies from its maximum to the minimum value, while the digital rate meter 22 (Fig. 2) measures the reciprocal of the rate of displacement of the piston 5 (Fig. 1), so that the recording means 24 records the signals coming from both code converters 23 and 26 in the reverse direction, i.e., the pen (not shown in the drawing) on the chart tape 27 (Fig. 4) must move both from top to bottom and from right to left.

For the herein-proposed capillary 3 (Fig. 1) and the chamber 4, the common logarithms of the pressure created by the spring 4 and of the rate of displacement of the piston 5 are scaled to the logarithmic values of the shearing stress and the rate of shear. The chart tape 27 (Fig. 4) of the recording means 24, allowing for the diameter and the capillary 3 (Fig. 1), therefore provides the curve 28 (Fig. 4) which is in fact the flow curve of the material to be investigated (Fig. 1).

The present capillary viscometer is designed to measure and automatically record in the log-log scale the flow curves which characterize viscous properties of elastomers, melts and concentrated solutions of polymeric and disperse systems. The measurements are carried out under isothermal conditions at a desired temperature.

Specifications

1. Apparent viscosity	$150-5.10^5$ poise
2. Shear stress	$5.10^2-5.10^5$ dyne/cm^2 at $\dfrac{\tau_{max}}{\tau_{min}}=10^2$
3. Rate of shear	$D=1.10^2-5.10^4$ sec^{-1} at $\dfrac{D_{max}}{D_{min}}=10^5$
4. Range of temperature in the chamber	-50 to $+250$°C
5. Principal errors:	
(a) shear stress	$\Delta\tau=\pm 0.015-0.002$
(b) rate of shear	$D=\pm 0.035-0.02 \, . \, D_{max}$
6. Amount of the material required to charge the chamber	7.5 cm^3
7. Scale of recording—logarithmic along both coordinate axes	
8. Weight of the device 120 kg	
9. Floor space occupied 830×470 mm^2	

WHAT WE CLAIM IS:—

1. A capillary viscometer comprising a spring, means for preliminarily compressing the spring, the spring being arranged to act on a piston so as, in use, to force material to be investigated out of a chamber through a capillary, a transducer for converting the displacement of the piston into an output signal, and means for converting the output signal into a voltage proportional to the common logarithm of the rate of displacement of the piston and into a voltage proportional to the common logarithm of the pressure created by the spring, the converting means being connected to a recording means for recording the size of the voltages.

2. A viscometer as claimed in Claim 1, wherein the transducer comprises an optical grating-type transducer.

3. A viscometer as claimed in Claim 1 or 2, wherein the means for converting the output signal from the transducer comprises a digital meter connected to the transducer for recording the rate of displacement of the piston, a code converter connected to the outputs of the meter and to an input of the recording means for converting the code from the digital meter into a voltage proportional to the common logarithm of the rate of displacement of the piston, a counter for counting pulses proportional in numbers to the displacement of the piston and connected to the transducer, and a code converter for convert-

ing the code from the counter into a voltage proportional to the common logarithm of the pressure created by the spring, connected to the outputs of the counter and to another input of the recording means, the recording means being arranged to produce a representation of a flow curve on its chart tape.

4. A capillary viscometer substantially as hereinbefore described with reference to the accompanying drawings.

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1463870 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

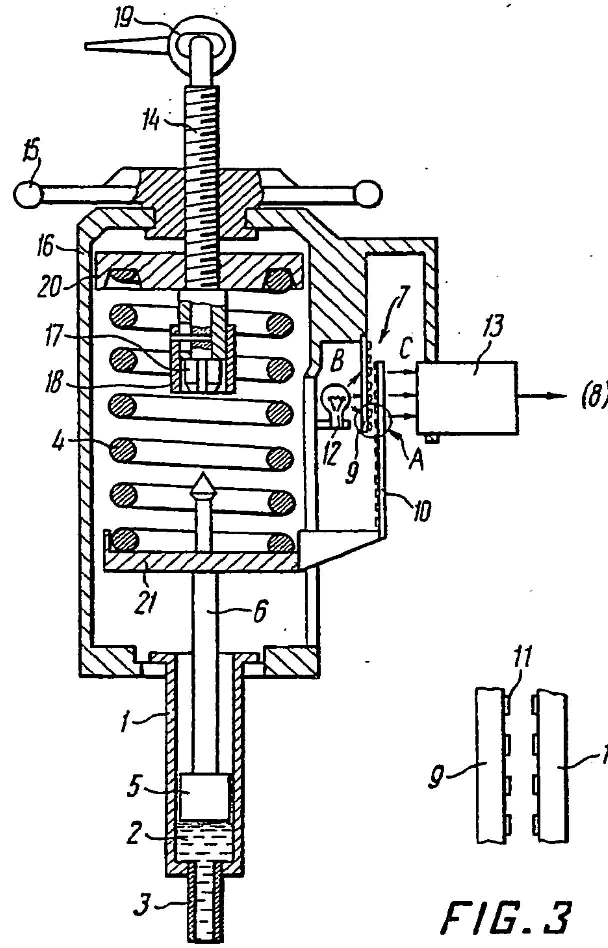

FIG.1

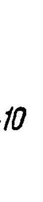

FIG.3

1463870 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

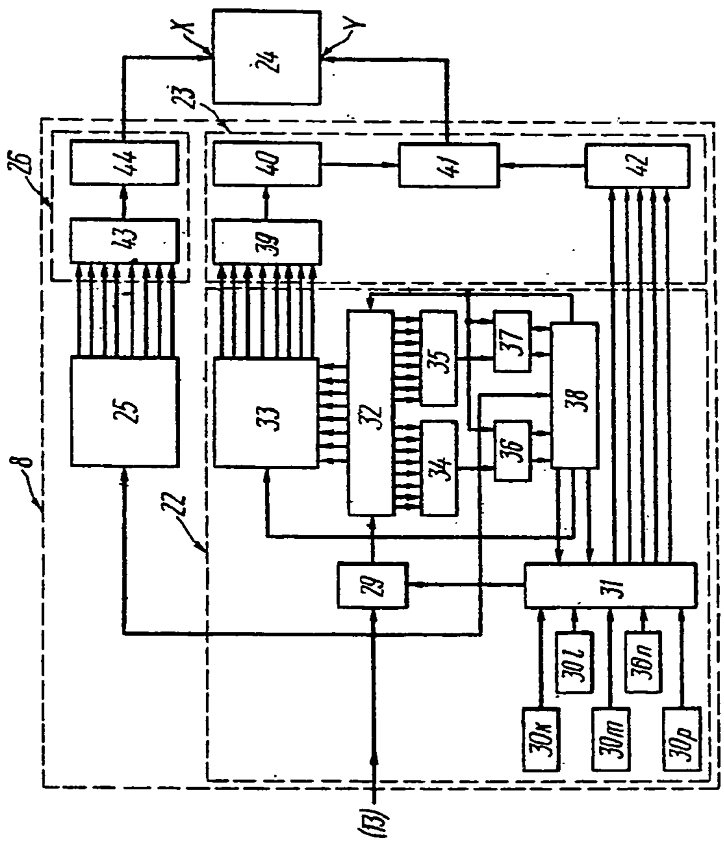

FIG. 2

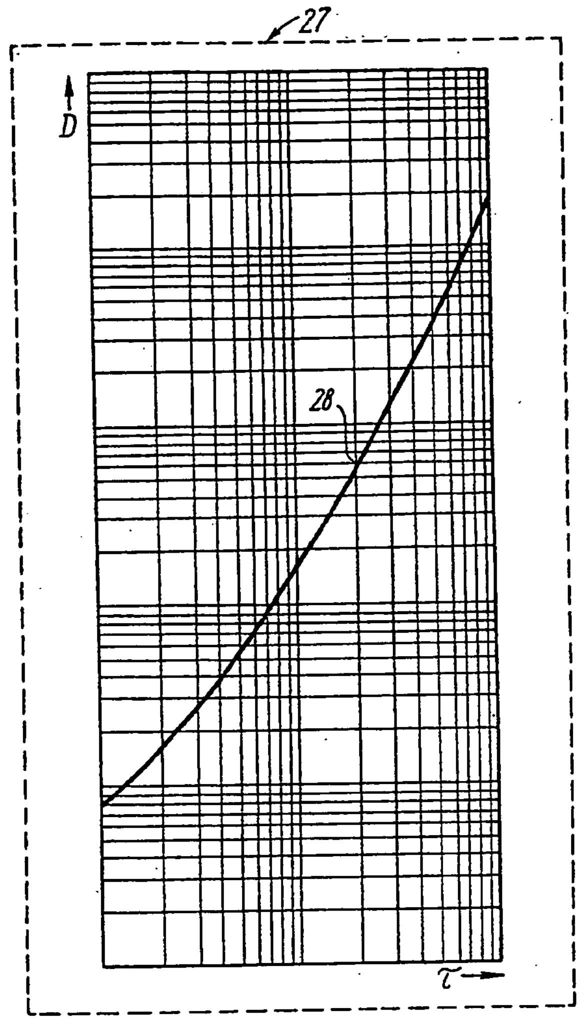

FIG. 4